



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(54) Title:</b> ALKYL POLYGLYCOSIDES IN TEXTILE SCOUR/BLEACH PROCESSING  <b>(57) Abstract</b>  A process for bleaching and scouring unfinished textile materials by adding to an aqueous alkaline peroxide bath an effective amount of a surfactant composition containing: (a) from about 94.0 to about 6.0 % by weight of an alkyl polyglycoside having the general formula (I): RO(Z) <sub>a</sub> wherein R is a monovalent organic radical having from about 8 to about 16 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6, (b) from about 6 to about 94 % by weight of a blend of alkoxyated branched C <sub>8</sub> -C <sub>15</sub> alcohols, and (c) the remainder water, all weights being based on the weight of the composition; and then contacting the textile materials with the bath.		

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## ALKYL POLYGLYCOSIDES IN TEXTILE SCOUR/BLEACH PROCESSING

Field of the Invention

The present invention generally relates to a process for scouring and bleaching textile materials. More particularly, by combining an alkyl polyglycoside with a blend of alkoxyated branched alcohols, a synergistic scouring and bleaching effect is realized.

Background of the Invention

Textile materials are among the most ubiquitous in society. They provide shelter and protection from the environment in the form of apparel, and comfort and decoration in the form of household textiles, such as sheets, upholstery, carpeting, drapery and wall covering, and they have a variety of industrial functions, such as tire reinforcement, tenting, filter media, conveyor belts, insulation, etc.

Textile materials are produced from fibers (finite lengths) and filaments (continuous lengths) by a variety of processes to form woven, knitted and nonwoven (felt-like) fabrics. In the case of woven and knitted fabrics, the fibers and filaments are formed into intermediate continuous-length structures known as yarns, which are interlaced by weaving or interlooped by knitting into planar-flexible sheetlike structures known as fabrics. Nonwoven fabrics are formed directly from fibers and filaments by chemically or physically bonding or interlocking fibers that have been arranged in a planar configuration.

Textile fibers are classified into two main categories, man-made and natural. Man-made fibers are formed by extrusion processes known as melt-dry, or wet spinning. The spinning or extrusion of filaments is normally followed by an operation known as drawing. In this step, the newly formed filaments are irreversibly extended and stabilized by setting or crystallization processes.

With the exception of silk, naturally occurring fibers have finite lengths and generally require several cleaning and purification steps prior to processing into yarns and fabrics.

There are a number of finishing processes that textile fibers are subjected to after their formation. The two with which the present invention is mostly concerned are scouring and bleaching. Scouring refers to the removal of

sizing materials, lubricants and other impurities which are contained in and/or adhere to the fibers during their formation. These various impurities must be removed so that the textile fibers may be further processed. Another  
5 finishing process is bleaching whereby a white color is imparted to the fabric. This bleaching step also enhances the absorbency of the fiber materials in preparation for the application of other finishing processes as well as the removal of any residual impurities left over from the  
10 scouring process.

Both the scouring and bleaching processes are performed under extremely alkaline conditions using high concentrations of peroxide and/or caustic soda, and at high temperatures. Due to the extremely hot and alkaline  
15 environment, there is a need for a textile scouring and bleaching composition which is stable under these circumstances, while at the same time having low levels of foam formation under high agitation. Moreover, as a result of the current degree of enhanced consciousness with  
20 respect to the protection of our environment, the composition employed should be highly biodegradable as well.

Thus, it is primary object of this invention to provide a more effective means of scouring and bleaching  
25 textile fibers in an environmentally safe manner.

#### Summary of the Invention

Other than in the operating examples, or where

otherwise indicated, all numbers expressing quantities of ingredients or reaction conditions used herein are to be understood as modified in all instances by the term "about".

5 Briefly stated, the present invention is directed to a process for scouring and bleaching textile materials comprising adding to an aqueous peroxide bleaching bath an effective amount of a scouring and bleaching surfactant composition comprising:

10 (a) from about 6.0 to about 94.0% by weight of an alkyl polyglycoside having the general formula I



wherein R is a monovalent organic radical having from about 8 to about 16 carbon atoms; Z is a saccharide residue  
15 having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6,

(b) from about 94.0 to about 6.0% by weight of a blend of alkoxyated C<sub>8</sub>-C<sub>15</sub> branched alcohols, and

(c) the remainder water, all weights being based on  
20 the weight of the composition, and then contacting said textile materials with said bath.

#### Brief Description of the Drawings

Fig. 1 is a bar graph illustrating the degree of foam generated by various blends of APG®600, Trycol 5943 and  
25 Trycol 6720, by measuring the height of foam formed after 2 minutes, at a temperature of about 120°F.

Fig. 2 is a bar graph illustrating the effects of various blends of APG®600, Trycol 5943 and Trycol 6720 on caustic stability, represented by sodium hydroxide content, at a temperature of about 160°F.

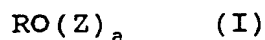
Fig. 3 is a bar graph illustrating the effects of various blends of APG®600, Trycol 5943 and Trycol 6720 on Draves wetting speed using a cotton substrate.

#### Description of the Invention

It has surprisingly been found that an exceedingly synergistic scouring and bleaching effect can be obtained for a wide variety of textile materials when combining the alkyl polyglycoside surfactant of this invention with a blend of branched alcohols that have been alkoxyated.

The aqueous peroxide bleaching bath typically contains finishing components present in an amount of from about 5.0 to about 20.0% by weight, based on weight of the bath. These finishing components include an alkali material, caustic soda, chelating agents and a surface-active material such as a surfactant. It is preferred that the composition be phosphate-free and contain no phenols.

The alkyl polyglycoside of the present invention is of the general formula I:



wherein R is a monovalent organic radical having from about 8 to about 16 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6.

The alkyl polyglycosides which can be used according to the invention preferably contain from about 12 to about 16 carbon atoms. These alkyl polyglycosides are commercially available, for example, as GLUCOPON® 325 and  
5 GLUCOPON® 600 from Henkel Corporation, Ambler, PA.

The alkyl polyglycosides have a dipole moment in the range of about 1.4 to about 1.7, and preferably about 1.4. The pH of the alkyl polyglycoside is preferably in the range of about 8 to about 9. The percent actives of the  
10 alkyl polyglycosides employed in the present invention is in the range of about 40 to about 70, and preferably about 50%.

The alkoxyated branched alcohols of the present invention are the condensation products of organic C<sub>8</sub>-C<sub>15</sub>  
15 alcohols, preferably C<sub>10</sub>-C<sub>13</sub> alcohols such as isodecyl alcohol and tridecyl alcohol, with from about 6 to about 12 moles of ethylene oxide and from about 4 to about 8 moles, preferably about 6 moles, of propylene oxide, per mole of alcohol. The alkoxyated branched alcohols preferably have  
20 an HLB value of from about 6 to about 15. Most preferably, the alkoxyated branched alcohols of the invention comprise a mixture of a tridecyl alcohol ethoxylated with about 12 moles of ethylene oxide per mole of alcohol and an alkoxyated isodecyl alcohol containing about 6 moles of  
25 ethylene oxide and about 6 moles of propylene oxide. A tridecyl alcohol ethoxylated with about 12 moles of ethylene oxide is commercially available under the tradename TRYCOL® 5943 from Henkel Corporation, Textiles



Division, Charlotte, N.C. An alkoxyated isodecyl alcohol containing about 6 moles of ethylene oxide and about 6 moles of propylene oxide is commercially available under the tradename TRYCOL® 6720 from Henkel Corporation, Textiles Division, Charlotte, N.C.

The amount of alkyl polyglycoside and alkoxyated branched alcohols to be used should be sufficient to effectively wet, i.e., impregnate and bleach the textile substrate and thus scour the substrate and improve the bleaching properties of the bath. The types of substrates which will be treated with the bleaching composition will vary, but will include articles of apparel made of cotton and polyester/cotton woven and knit goods.

In a particularly preferred embodiment of the present invention, the composition to be added to the aqueous peroxide bleaching bath contains from about 20 to 60% by weight of the alkyl polyglycoside and from about 80 to 40% by weight of the alkoxyated branched alcohols. Also, the amount of the scouring and bleaching surfactant composition to be added to the bath is typically in the range from 0.1 to 1.0% by weight, based on the weight of the bath, and preferably from about 0.1 to about 0.2% by weight.

In addition, the ratio by weight of alkyl polyglycoside to tridecyl alcohol ethoxylate may be from about 2 to about 1, preferably from about 1.5 to about 1, and most preferably is about 1:1. The ratio by weight of alkyl polyglycoside to alkoxyated isodecyl alcohol may be from about 2 to about 1, preferably from about 1 to about

1, and most preferably is about 0.5:1.

It has been found that the composition containing alkyl polyglycoside and the mixture of alkoxyated branched alcohols used in the invention provides a caustic stable,  
5 low foaming, phosphate-free scouring and bleaching aid in processing textile materials.

Generally, the continuous scouring and bleaching process for cotton and polyester/cotton woven goods is carried out in two separate steps, and in either rope or  
10 open-width form using an exhaust bath..

In the scour step, the pH of the alkaline aqueous impregnation (exhaust) bath is most preferably between about 11 and 12.0 and the temperature of the bath is preferably between about 120 and 200°F, and most preferably  
15 about 160°F. The desized fabric is immersed in the alkaline scour bath and squeezed, by pad rolls, to a wet pickup of from about 90 to 110% owg (i.e. on the weight of the goods). The treated fabric is then placed in a steam chamber for about 15 minutes (to simulate the open-width  
20 process) or about 60 minutes (to simulate the rope process). The steamed fabric is then washed in water at a temperature from about 180 to 200°F, for about 60 to 90 seconds.

In the bleaching step, the pH of the alkaline aqueous  
25 bleaching exhaust bath is preferably between about 10.8 and about 11.2, and most preferably about 11.0. The temperature of the aqueous bath is preferably between about 75 to about 120°F, and most preferably about 100°F. The

scoured fabric is then impregnated with the aqueous alkaline bleaching bath in the same manner as in the scouring process above. The treated fabric is then steamed in the same manner as disclosed above for the scouring process. The steamed fabric is then washed in water at a temperature of about 180 to 200°F, for approximately 60 to 90 seconds, followed by a cold rinse in water containing acetic acid to neutralize any residual alkali that may be present. The washed fabric is then dried at about 250°F.

The continuous scouring and bleaching process for cotton and polyester/cotton knit goods may also be performed per the one-bath under liquor method. The pH of the bath is preferably between about 10.8 and 11.2 , and most preferably about 11.0. The temperature of the aqueous bath is preferably between about 180 and about 200°F, and most preferably about 185°F with the dwell time being about 30 to 45 minutes. This step is followed by washing at about 160 to 185°F for approximately 15 to 20 minutes. It should be noted, however, that the pH and temperature ranges are dependent on a number of variables including the type of substrate being treated.

Another method of applying the aqueous bath is known as a padding operation, i.e., using a padding bath, whereby the bath is padded or blotted onto the substrate. This operation is very similar to that of the continuous dyeing operation since the substrate is mechanically carried into and out of the padding apparatus. When employing the padding bath, the aqueous scouring and/or bleaching bath

will have a pH in the range of about 11.0 to 12.0, and preferably in the range of about 10.8 to 11.2. It should be noted, however, that either the exhaust bath or padding bath may be employed to scour and/or bleach the fabric when  
5 using the scouring and bleaching surfactant composition in the present invention.

The present invention will be better understood when read in light of the following examples. In the following examples, parts and percentages are by weight and the  
10 temperatures are in degrees Celsius.

In the examples, the following five test methods were used to evaluate the effectiveness of the scouring and bleaching composition:

I. Whiteness Measurement:

15 Procedure

(1) Desized, scoured cotton or polyester/cotton woven goods were immersed in the aqueous bleaching and scouring composition and squeezed, by pad rolls, to a wet pickup of 90-110% owg (on the weight of the goods).

20 (2) The treated fabric was then placed in a steamer for about 15 minutes (to simulate open-width bleach) or 60 minutes (to simulate rope bleach).

(3) The steamed fabric was then washed in water at about 180 to 200°F for approximately 60 seconds. This  
25 washing process was then repeated and followed by a cold rinse in water containing acetic acid to neutralize any residual alkalinity.

(4) The washed fabric was then dried at about 250°F.

Evaluation Method For Whiteness

The bleached and scoured fabric was then evaluated using a Gardner reflectometer to measure whiteness.

5     2. Foam Measurement:

Bleach baths (see Comparative Examples) were tested for foam propensity using either a scour bath formulation or bleach bath formulation. The test apparatus was a recirculating foam tester that sprays the bath onto the surface of the bath held in the reservoir. For this particular test, the foam measurement was performed using a recirculation pump foam tester at 70 volts, at 120°F for 30 seconds.

3. Wetting Property Measurement:

15     Draves Test: Performed using AATCC-17-1989.

4. Cleanliness (%Extractibles):

Performed using: (1) Enzyme and water, and (2) perchloroethylene (AATCC-97-1989).

5. Caustic Stability:

20     Performed using 10g/l test sample in 50% caustic soda solution, at 160°F.

EXAMPLE 1

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
5	(a) GLUCOPON 600 UP	13.33
	(b) TRYCOL 5943	13.33
	(c) TRYCOL 6720	13.33
	(d) water	<u>60.01</u>
		100.00

10

EXAMPLE 2

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	20.0
15	(b) TRYCOL 5943	20.0
	(c) TRYCOL 6720	0.0
	(d) water	<u>60.0</u>
		100.0

EXAMPLE 3

20 A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	0.0
	(b) TRYCOL 5943	20.0
25	(c) TRYCOL 6720	20.0
	(d) water	<u>60.0</u>
		100.0

EXAMPLE 4

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
5	(a) GLUCOPON 600 UP	20.0
	(b) TRYCOL 5943	0.0
	(c) TRYCOL 6720	20.0
	(d) water	<u>60.0</u>
		100.0

10

EXAMPLE 5

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	9.9
15	(b) TRYCOL 5943	8.0
	(c) TRYCOL 6720	22.1
	(d) water	<u>60.0</u>
		100.0

20

EXAMPLE 6

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	22.1
25	(b) TRYCOL 5943	8.0
	(c) TRYCOL 6720	9.9
	(d) water	<u>60.0</u>
		100.0

EXAMPLE 7

A scouring and bleaching surfactant composition was prepared having the following components:

5	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	8.0
	(b) TRYCOL 5943	9.9
	(c) TRYCOL 6720	22.1
	(d) water	<u>60.0</u>
10		100.0

EXAMPLE 8

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
15	(a) GLUCOPON 600 UP	0.0
	(b) TRYCOL 5943	0.0
	(c) TRYCOL 6720	40.0
	(d) water	<u>60.0</u>
		100.0

EXAMPLE 9

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	0.0
25	(b) TRYCOL 5943	40.0
	(c) TRYCOL 6720	0.0
	(d) water	<u>60.0</u>
		100.0



EXAMPLE 10

A scouring and bleaching surfactant composition was prepared having the following components:

5	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	40.0
	(b) TRYCOL 5943	0.0
	(c) TRYCOL 6720	0.0
	(d) water	<u>60.0</u>
10		100.0

EXAMPLES 1-10

Examples 1-10 were used to scour and bleach cotton substrates by impregnation with an aqueous peroxide bleaching bath having a pH of about 11.0, followed by steaming at a temperature of about 98°C for a period of about 60 minutes.

Each sample was evaluated per the above stated testing methods for whiteness, foam formation, alkali stability, wetting properties and cleanliness, i.e., % Extractibles, the results being set forth in Table I.

TABLE I

EXAMPLE	FOAM PRODUCED	WHITENESS RATING	DRAVES TEST (SEC.)	(%) EXTRAC- TIBLES	CAUSTIC STABILITY (% OF 50% NaOH)
1	high	76.12	6.4	1.01	60
2	very high	75.73	14.3	0.35	80
3	high	76.2	4.9	0.57	2.5
4	meduim	77.15	9.8	0.91	70
5	low	74.6	5.7	0.43	50
6	very high	75.1	11.3	0.41	70
7	high	75	11.3	0.68	50
8	very low	72.73	6.5	0.59	1
9	high	75.1	8.5	0.47	40
10	very high	71.82	14.5	0.54	99

From the foregoing evaluation results, it can be seen that the bleaching and scouring process of this invention synergistically provides excellent whiteness to fabric materials, low residual impurities, is stable in high concentrations of strongly alkaline materials at high temperatures, and is low-foaming under high agitation conditions.

What is claimed is:

1. A process for bleaching and scouring textile materials comprising adding to an aqueous alkaline peroxide bath an effective amount of a surfactant composition  
5 comprising:

(a) from about 94.0 to about 6.0% by weight of an alkyl polyglycoside having the general formula I



wherein R is a monovalent organic radical having from about  
10 8 to about 16 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6,

(b) from about 6 to about 94% by weight of a blend of alkoxyated branched C<sub>8</sub>-C<sub>15</sub> alcohols, and

15 (c) the remainder water, all weights being based on the weight of the composition;  
contacting said textile materials with said bath and then steaming said textile materials.

2. The process of claim 1 wherein said alkyl  
20 polyglycoside has from about 12 to about 16 carbon atoms.

3. The process of claim 1 wherein said alkyl polyglycoside has a percent actives of about 50%.

4. The process of claim 1 wherein said alkoxyated alcohols contain from about 6 to about 12 moles of ethylene  
25 oxide and from about 4 to about 8 moles of propylene oxide per mole of said alcohol.

5. The process of claim 4 wherein said alkoxyated

alcohols have an HLB value of from about 6 to about 15.

6. The process of claim 1 wherein said blend of alkoxyated alcohols comprises a mixture of a tridecyl alcohol ethoxylated with about 12 moles of ethylene oxide  
5 per mole of alcohol and an isodecyl alcohol containing about 6 moles of ethylene oxide and about 6 moles of propylene oxide.

7. The process of claim 1 wherein said textile materials comprise cotton or a blend of polyester and cotton.

10 8. The process of claim 1 wherein from about 0.1 to about 1.0% by weight of said surfactant composition is added to said aqueous bath, based on the weight of said bath.

9. The process of claim 1 wherein from about 0.1 to about 0.2% by weight of said surfactant composition is added to  
15 said aqueous bath, based on the weight of said bath.

10. The process of claim 1 wherein said surfactant composition contains from about 20 to about 60% by weight of said component (a) and from about 80 to about 40% by weight of said component (b).

20 11. The process of claim 1 wherein said bath further contains from about 5.0 to about 20.0% by weight of additional finishing components, based on the weight of said bath.

12. The process of claim 11 wherein said finishing  
25 components are selected from sodium hydroxide, sodium silicate, and mixtures thereof.

13. The process of claim 1 wherein said bath is a bleaching exhaust bath.

14. The process of claim 1 wherein said bath is a scouring exhaust bath.

15. The process of claim 1 wherein said bath is both a scouring and bleaching exhaust bath.

5 16. The process of claim 1 wherein said bath is a bleaching padding bath.

17. The process of claim 1 wherein said bath is a scouring padding bath.

10 18. The process of claim 1 wherein said bath is both a scouring and bleaching padding bath.

19. The process of claim 15 wherein said scouring and bleaching exhaust bath is at a temperature in the range of about 180°F. to about 200°F.

15 20. The process of claim 1 wherein said bath has a pH of about 10.8 to about 11.2.

21. A surfactant composition for use in bleaching and scouring textile materials while controlling foam generation, said composition comprising:

20 (a) from about 94.0 to about 6.0% by weight of an alkyl polyglycoside having the general formula I



wherein R is a monovalent organic radical having from about 8 to about 16 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a  
25 value from about 1 to about 6,

(b) from about 6 to about 94% by weight of a blend of alkoxylated branched C<sub>8</sub>-C<sub>15</sub> alcohols, and

(c) the remainder water, all weights being based on the weight of the composition.

22. A composition as in claim 21 wherein said alkyl polyglycoside has from about 12 to about 16 carbon atoms.

5 23. A composition as in claim 21 wherein said alkyl polyglycoside has a percent actives of about 50%.

24. A composition as in claim 21 wherein said alkoxyated alcohols contain from about 6 to about 12 moles of ethylene oxide and from about 4 to about 8 moles of propylene oxide  
10 per mole of said alcohol.

25. A composition as in claim 21 wherein said alkoxyated alcohols have an HLB value of from about 6 to about 15.

26. A composition as in claim 21 containing from about 20% to about 60% by weight of said component (a) and from about  
15 80% to about 40% by weight of said component (b).

27. A composition as in claim 21 wherein said blend of alkoxyated alcohols comprises a mixture of a tridecyl alcohol ethoxylated with about 12 moles of ethylene oxide per mole of alcohol and an isodecyl alcohol containing  
20 about 6 moles of ethylene oxide and about 6 moles of propylene oxide.

28. A composition as in claim 21 further containing from about 5.0% to about 20.0% by weight of additional finishing components, based on the weight of said composition.

25 29. A composition as in claim 28 wherein said finishing components are selected from sodium hydroxide, sodium silicate, and mixtures thereof.

APG® SURFACTANTS FOAMING

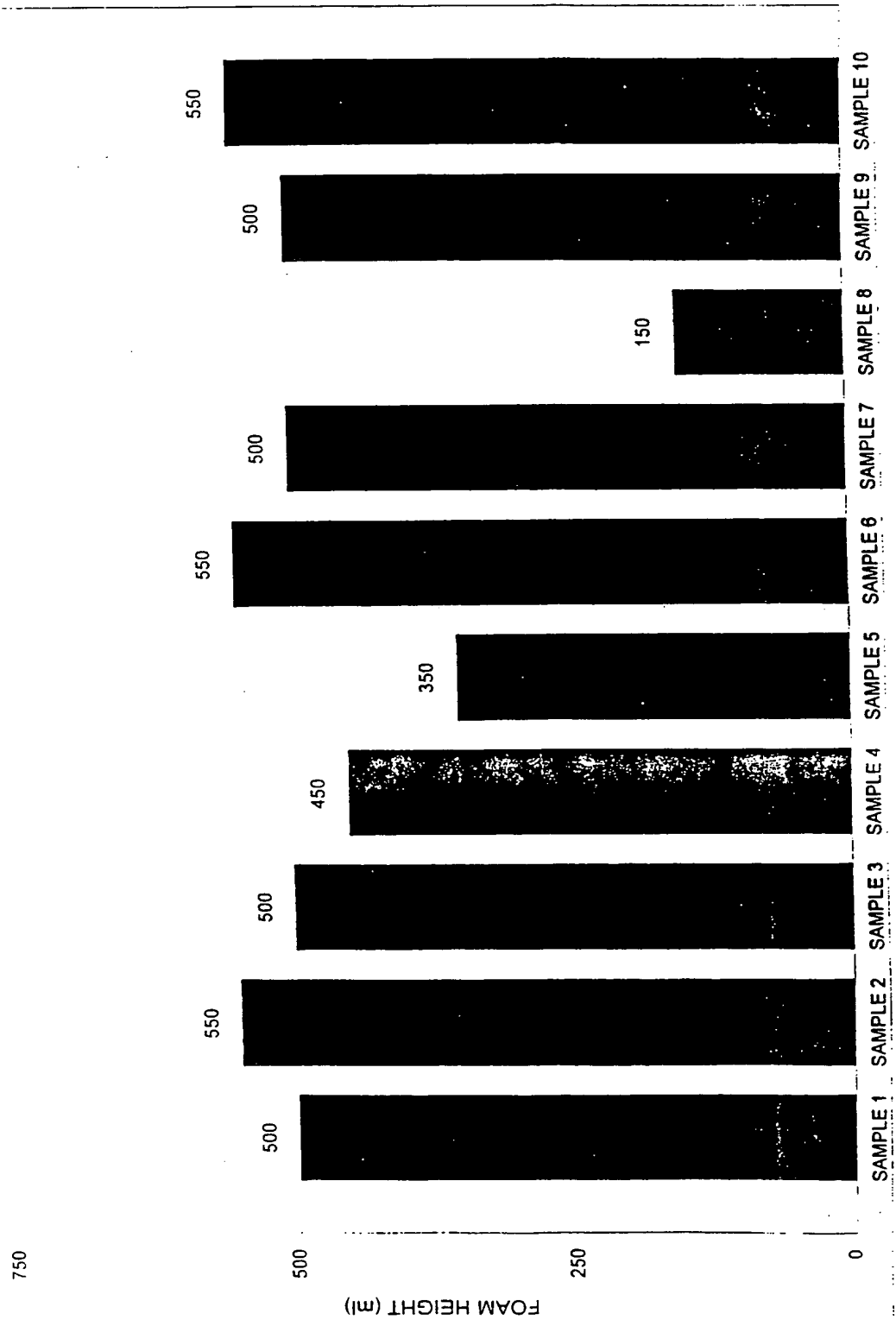


FIG. 2

APG® SURFACTANTS CAUSTIC STABILITY

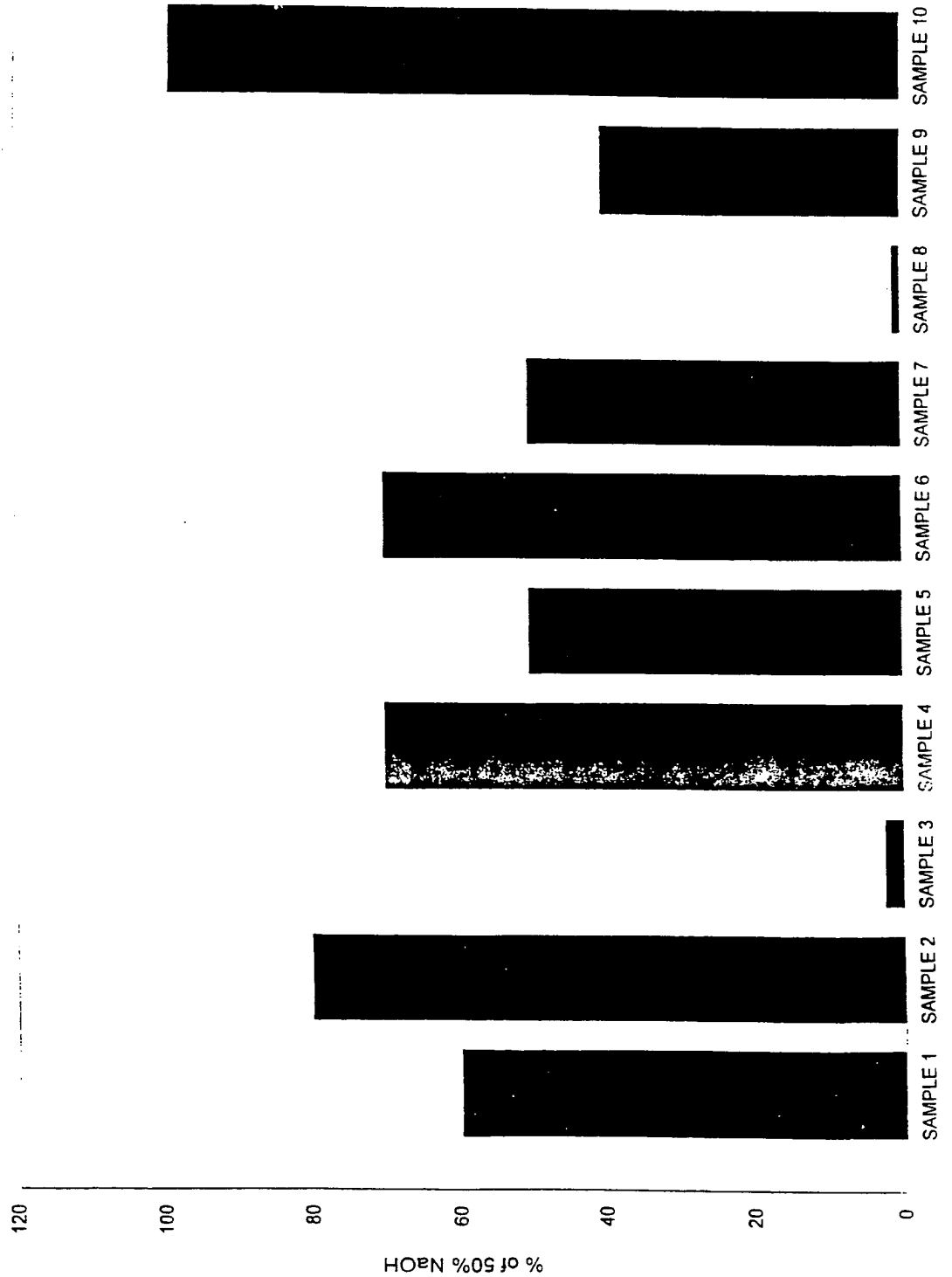
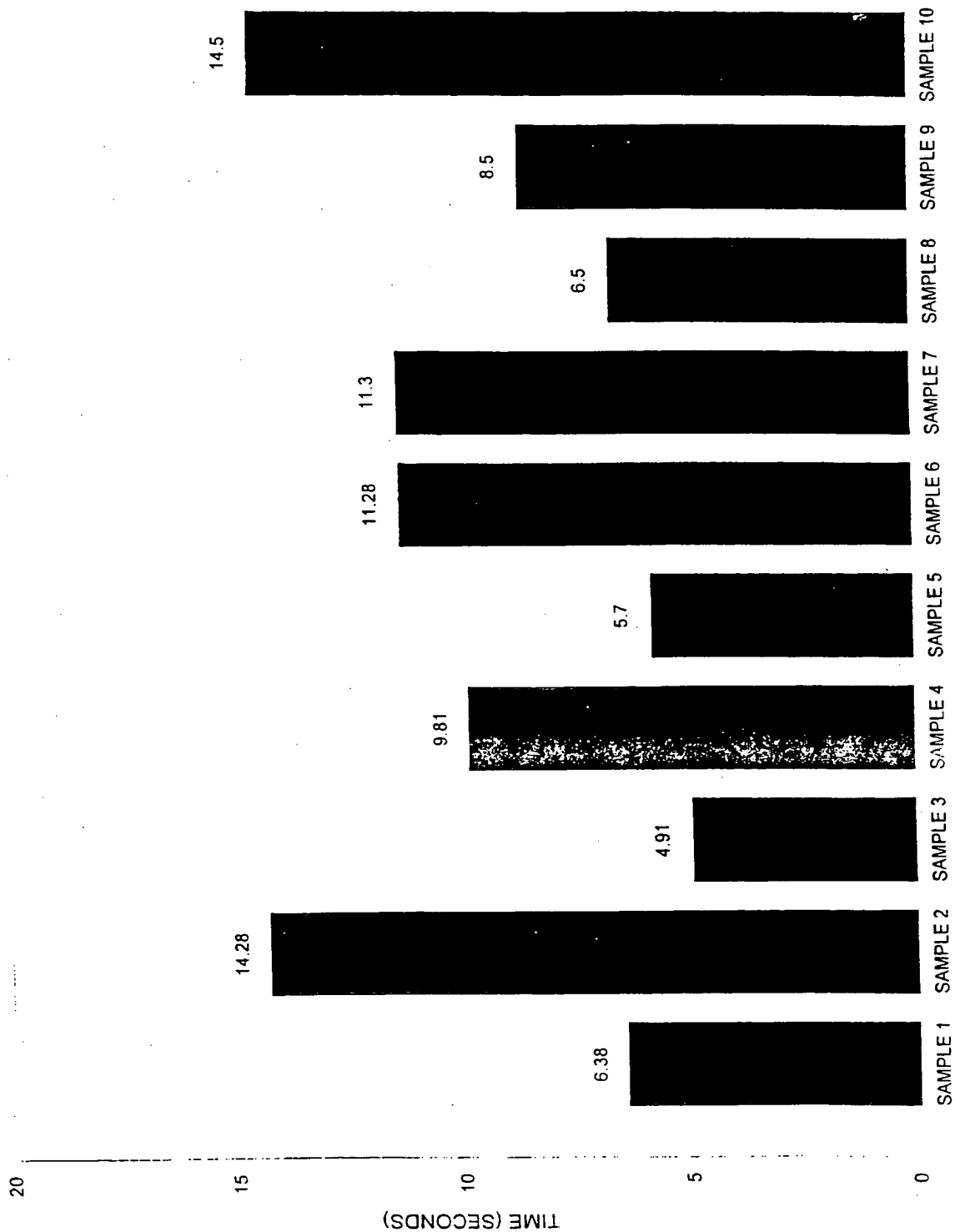




FIG. 3

APG® SURFACTANTS WETTING SPEED



## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US98/08585

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : D06L 3/02; C11D 1/66, 1/72, 1/722

US CL : 8/111, 139; 510/535, 303, 470, 506, 340; 252/351

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 8/111, 139; 510/535, 303, 470, 506, 340; 252/351

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4,483,780 A (LLENADO) 20 November 1984 (20-11-84), column 1, lines 37-57; column 2, lines 50-68; column 5, lines 5-35; and column 6, lines 1-38.	21-23, 25, 26, 28, 29
X	US 5,047,168 A (BROZE et al) 10 September 1991 (10-09-91), column 5, line 61 through column 6, line 65; column 8, lines 10-24; column 9, lines 49-68; and column 12, lines 1-41.	21-26, 28, 29
A	US 5,527,362 A (COLE et al) 18 June 1996 (18-06-96).	1-29
A	US 5,542,950 A (COLE et al) 06 August 1996 (06-08-96).	1-29



Further documents are listed in the continuation of Box C.



See patent family annex.

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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/US98/08585 <b>(22) International Filing Date:</b> 1 May 1998 (01.05.98)  <b>(30) Priority Data:</b> 08/850,900      1 May 1997 (01.05.97)      US  <b>(71) Applicant:</b> HENKEL CORPORATION [US/US]; Suite 200, 2500 Renaissance Boulevard, Gulph Mills, PA 19406 (US).  <b>(72) Inventors:</b> FRANCOIS, Brian, C.; 8902 Shorehaven Court, Charlotte, NC 28269 (US). GLENN, Susan, C.; 9915 Wolf Creek Trail, Charlotte, NC 28269 (US). COLE, Howard; 5037 Virginian Lane, Charlotte, NC 28226 (US).  <b>(74) Agent:</b> DRACH, John, E.; Henkel Corporation, Suite 200, 2500 Renaissance Boulevard, Gulph Mills, PA 19406 (US).		<b>(81) Designated States:</b> AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> ALKYL POLYGLYCOSIDES IN TEXTILE SCOUR/BLEACH PROCESSING  <b>(57) Abstract</b> <p>A process for bleaching and scouring unfinished textile materials by adding to an aqueous alkaline peroxide bath an effective amount of a surfactant composition containing: (a) from about 94.0 to about 6.0 % by weight of an alkyl polyglycoside having the general formula (I): RO(Z)<sub>a</sub> wherein R is a monovalent organic radical having from about 8 to about 16 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6, (b) from about 6 to about 94 % by weight of a blend of alkoxylated branched C<sub>8</sub>-C<sub>15</sub> alcohols, and (c) the remainder water, all weights being based on the weight of the composition; and then contacting the textile materials with the bath.</p>		

\*(Referred to in PCT Gazette No. 12/1999, Section II) \*\*(Referred to in PCT Gazette No. 17/1999, Section II)

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## ALKYL POLYGLYCOSIDES IN TEXTILE SCOUR/BLEACH PROCESSING

Field of the Invention

The present invention generally relates to a process for scouring and bleaching textile materials. More particularly, by combining an alkyl polyglycoside with a blend of alkoxyated branched alcohols, a synergistic scouring and bleaching effect is realized.

Background of the Invention

Textile materials are among the most ubiquitous in society. They provide shelter and protection from the environment in the form of apparel, and comfort and decoration in the form of household textiles, such as sheets, upholstery, carpeting, drapery and wall covering, and they have a variety of industrial functions, such as tire reinforcement, tenting, filter media, conveyor belts, insulation, etc.

Textile materials are produced from fibers (finite lengths) and filaments (continuous lengths) by a variety of processes to form woven, knitted and nonwoven (felt-like) fabrics. In the case of woven and knitted fabrics, the fibers and filaments are formed into intermediate continuous-length structures known as yarns, which are interlaced by weaving or interlooped by knitting into planar-flexible sheetlike structures known as fabrics. Nonwoven fabrics are formed directly from fibers and filaments by chemically or physically bonding or interlocking fibers that have been arranged in a planar configuration.

Textile fibers are classified into two main categories, man-made and natural. Man-made fibers are formed by extrusion processes known as melt-dry, or wet spinning. The spinning or extrusion of filaments is normally followed by an operation known as drawing. In this step, the newly formed filaments are irreversibly extended and stabilized by setting or crystallization processes.

With the exception of silk, naturally occurring fibers have finite lengths and generally require several cleaning and purification steps prior to processing into yarns and fabrics.

There are a number of finishing processes that textile fibers are subjected to after their formation. The two with which the present invention is mostly concerned are scouring and bleaching. Scouring refers to the removal of

sizing materials, lubricants and other impurities which are contained in and/or adhere to the fibers during their formation. These various impurities must be removed so that the textile fibers may be further processed. Another  
5 finishing process is bleaching whereby a white color is imparted to the fabric. This bleaching step also enhances the absorbency of the fiber materials in preparation for the application of other finishing processes as well as the removal of any residual impurities left over from the  
10 scouring process.

Both the scouring and bleaching processes are performed under extremely alkaline conditions using high concentrations of peroxide and/or caustic soda, and at high temperatures. Due to the extremely hot and alkaline  
15 environment, there is a need for a textile scouring and bleaching composition which is stable under these circumstances, while at the same time having low levels of foam formation under high agitation. Moreover, as a result of the current degree of enhanced consciousness with  
20 respect to the protection of our environment, the composition employed should be highly biodegradable as well.

Thus, it is primary object of this invention to provide a more effective means of scouring and bleaching  
25 textile fibers in an environmentally safe manner.

#### Summary of the Invention

Other than in the operating examples, or where

otherwise indicated, all numbers expressing quantities of ingredients or reaction conditions used herein are to be understood as modified in all instances by the term "about".

5 Briefly stated, the present invention is directed to a process for scouring and bleaching textile materials comprising adding to an aqueous peroxide bleaching bath an effective amount of a scouring and bleaching surfactant composition comprising:

10 (a) from about 6.0 to about 94.0% by weight of an alkyl polyglycoside having the general formula I



wherein R is a monovalent organic radical having from about 8 to about 16 carbon atoms; Z is a saccharide residue  
15 having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6,

(b) from about 94.0 to about 6.0% by weight of a blend of alkoxyated C<sub>8</sub>-C<sub>15</sub> branched alcohols, and

(c) the remainder water, all weights being based on  
20 the weight of the composition, and then contacting said textile materials with said bath.

#### Brief Description of the Drawings

Fig. 1 is a bar graph illustrating the degree of foam generated by various blends of APG®600, Trycol 5943 and  
25 Trycol 6720, by measuring the height of foam formed after 2 minutes, at a temperature of about 120°F.



Fig. 2 is a bar graph illustrating the effects of various blends of APG®600, Trycol 5943 and Trycol 6720 on caustic stability, represented by sodium hydroxide content, at a temperature of about 160°F.

5 Fig. 3 is a bar graph illustrating the effects of various blends of APG®600, Trycol 5943 and Trycol 6720 on Draves wetting speed using a cotton substrate.

#### Description of the Invention

10 It has surprisingly been found that an exceedingly synergistic scouring and bleaching effect can be obtained for a wide variety of textile materials when combining the alkyl polyglycoside surfactant of this invention with a blend of branched alcohols that have been alkoxyated.

15 The aqueous peroxide bleaching bath typically contains finishing components present in an amount of from about 5.0 to about 20.0% by weight, based on weight of the bath. These finishing components include an alkali material, caustic soda, chelating agents and a surface-active material such as a surfactant. It is preferred that the  
20 composition be phosphate-free and contain no phenols.

The alkyl polyglycoside of the present invention is of the general formula I:



25 wherein R is a monovalent organic radical having from about 8 to about 16 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6.

The alkyl polyglycosides which can be used according to the invention preferably contain from about 12 to about 16 carbon atoms. These alkyl polyglycosides are commercially available, for example, as GLUCOPON® 325 and  
5 GLUCOPON® 600 from Henkel Corporation, Ambler, PA.

The alkyl polyglycosides have a dipole moment in the range of about 1.4 to about 1.7, and preferably about 1.4. The pH of the alkyl polyglycoside is preferably in the range of about 8 to about 9. The percent actives of the  
10 alkyl polyglycosides employed in the present invention is in the range of about 40 to about 70, and preferably about 50%.

The alkoxyated branched alcohols of the present invention are the condensation products of organic  $C_8$ - $C_{15}$   
15 alcohols, preferably  $C_{10}$ - $C_{13}$  alcohols such as isodecyl alcohol and tridecyl alcohol, with from about 6 to about 12 moles of ethylene oxide and from about 4 to about 8 moles, preferably about 6 moles, of propylene oxide, per mole of alcohol. The alkoxyated branched alcohols preferably have  
20 an HLB value of from about 6 to about 15. Most preferably, the alkoxyated branched alcohols of the invention comprise a mixture of a tridecyl alcohol ethoxylated with about 12 moles of ethylene oxide per mole of alcohol and an alkoxyated isodecyl alcohol containing about 6 moles of  
25 ethylene oxide and about 6 moles of propylene oxide. A tridecyl alcohol ethoxylated with about 12 moles of ethylene oxide is commercially available under the tradename TRYCOL® 5943 from Henkel Corporation, Textiles

Division, Charlotte, N.C. An alkoxylated isodecyl alcohol containing about 6 moles of ethylene oxide and about 6 moles of propylene oxide is commercially available under the tradename TRYCOL® 6720 from Henkel Corporation, Textiles Division, Charlotte, N.C.

The amount of alkyl polyglycoside and alkoxylated branched alcohols to be used should be sufficient to effectively wet, i.e., impregnate and bleach the textile substrate and thus scour the substrate and improve the bleaching properties of the bath. The types of substrates which will be treated with the bleaching composition will vary, but will include articles of apparel made of cotton and polyester/cotton woven and knit goods.

In a particularly preferred embodiment of the present invention, the composition to be added to the aqueous peroxide bleaching bath contains from about 20 to 60% by weight of the alkyl polyglycoside and from about 80 to 40% by weight of the alkoxylated branched alcohols. Also, the amount of the scouring and bleaching surfactant composition to be added to the bath is typically in the range from 0.1 to 1.0% by weight, based on the weight of the bath, and preferably from about 0.1 to about 0.2% by weight.

In addition, the ratio by weight of alkyl polyglycoside to tridecyl alcohol ethoxylate may be from about 2 to about 1, preferably from about 1.5 to about 1, and most preferably is about 1:1. The ratio by weight of alkyl polyglycoside to alkoxylated isodecyl alcohol may be from about 2 to about 1, preferably from about 1 to about

1, and most preferably is about 0.5:1.

It has been found that the composition containing alkyl polyglycoside and the mixture of alkoxyated branched alcohols used in the invention provides a caustic stable, low foaming, phosphate-free scouring and bleaching aid in processing textile materials.

Generally, the continuous scouring and bleaching process for cotton and polyester/cotton woven goods is carried out in two separate steps, and in either rope or open-width form using an exhaust bath..

In the scour step, the pH of the alkaline aqueous impregnation (exhaust) bath is most preferably between about 11 and 12.0 and the temperature of the bath is preferably between about 120 and 200°F, and most preferably about 160°F. The desized fabric is immersed in the alkaline scour bath and squeezed, by pad rolls, to a wet pickup of from about 90 to 110% owg (i.e. on the weight of the goods). The treated fabric is then placed in a steam chamber for about 15 minutes (to simulate the open-width process) or about 60 minutes (to simulate the rope process). The steamed fabric is then washed in water at a temperature from about 180 to 200°F, for about 60 to 90 seconds.

In the bleaching step, the pH of the alkaline aqueous bleaching exhaust bath is preferably between about 10.8 and about 11.2, and most preferably about 11.0. The temperature of the aqueous bath is preferably between about 75 to about 120°F, and most preferably about 100°F. The

scoured fabric is then impregnated with the aqueous alkaline bleaching bath in the same manner as in the scouring process above. The treated fabric is then steamed in the same manner as disclosed above for the scouring process. The steamed fabric is then washed in water at a temperature of about 180 to 200°F, for approximately 60 to 90 seconds, followed by a cold rinse in water containing acetic acid to neutralize any residual alkali that may be present. The washed fabric is then dried at about 250°F.

The continuous scouring and bleaching process for cotton and polyester/cotton knit goods may also be performed per the one-bath under liquor method. The pH of the bath is preferably between about 10.8 and 11.2 , and most preferably about 11.0. The temperature of the aqueous bath is preferably between about 180 and about 200°F, and most preferably about 185°F with the dwell time being about 30 to 45 minutes. This step is followed by washing at about 160 to 185°F for approximately 15 to 20 minutes. It should be noted, however, that the pH and temperature ranges are dependent on a number of variables including the type of substrate being treated.

Another method of applying the aqueous bath is known as a padding operation, i.e., using a padding bath, whereby the bath is padded or blotted onto the substrate. This operation is very similar to that of the continuous dyeing operation since the substrate is mechanically carried into and out of the padding apparatus. When employing the padding bath, the aqueous scouring and/or bleaching bath

will have a pH in the range of about 11.0 to 12.0, and preferably in the range of about 10.8 to 11.2. It should be noted, however, that either the exhaust bath or padding bath may be employed to scour and/or bleach the fabric when using the scouring and bleaching surfactant composition in the present invention.

The present invention will be better understood when read in light of the following examples. In the following examples, parts and percentages are by weight and the temperatures are in degrees Celsius.

In the examples, the following five test methods were used to evaluate the effectiveness of the scouring and bleaching composition:

#### I. Whiteness Measurement:

##### Procedure

(1) Desized, scoured cotton or polyester/cotton woven goods were immersed in the aqueous bleaching and scouring composition and squeezed, by pad rolls, to a wet pickup of 90-110% owg (on the weight of the goods).

(2) The treated fabric was then placed in a steamer for about 15 minutes (to simulate open-width bleach) or 60 minutes (to simulate rope bleach).

(3) The steamed fabric was then washed in water at about 180 to 200°F for approximately 60 seconds. This washing process was then repeated and followed by a cold rinse in water containing acetic acid to neutralize any residual alkalinity.

(4) The washed fabric was then dried at about 250°F.

Evaluation Method For Whiteness

The bleached and scoured fabric was then evaluated using a Gardner reflectometer to measure whiteness.

5     2. Foam Measurement:

Bleach baths (see Comparative Examples) were tested for foam propensity using either a scour bath formulation or bleach bath formulation. The test apparatus was a recirculating foam tester that sprays the bath onto the  
10     surface of the bath held in the reservoir. For this particular test, the foam measurement was performed using a recirculation pump foam tester at 70 volts, at 120°F for 30 seconds.

3. Wetting Property Measurement:

15     Draves Test: Performed using AATCC-17-1989.

4. Cleanliness (%Extractibles):

Performed using: (1) Enzyme and water, and (2) perchloroethylene (AATCC-97-1989).

5. Caustic Stability:

20     Performed using 10g/l test sample in 50% caustic soda solution, at 160°F.

EXAMPLE 1

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
5	(a) GLUCOPON 600 UP	13.33
	(b) TRYCOL 5943	13.33
	(c) TRYCOL 6720	13.33
	(d) water	<u>60.01</u>
		100.00

10

EXAMPLE 2

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	20.0
15	(b) TRYCOL 5943	20.0
	(c) TRYCOL 6720	0.0
	(d) water	<u>60.0</u>
		100.0

EXAMPLE 3

20 A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	0.0
	(b) TRYCOL 5943	20.0
25	(c) TRYCOL 6720	20.0
	(d) water	<u>60.0</u>
		100.0



EXAMPLE 4

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
5	(a) GLUCOPON 600 UP	20.0
	(b) TRYCOL 5943	0.0
	(c) TRYCOL 6720	20.0
	(d) water	<u>60.0</u>
		100.0

10

EXAMPLE 5

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	9.9
15	(b) TRYCOL 5943	8.0
	(c) TRYCOL 6720	22.1
	(d) water	<u>60.0</u>
		100.0

20

EXAMPLE 6

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	22.1
25	(b) TRYCOL 5943	8.0
	(c) TRYCOL 6720	9.9
	(d) water	<u>60.0</u>
		100.0

EXAMPLE 7

A scouring and bleaching surfactant composition was prepared having the following components:

5	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	8.0
	(b) TRYCOL 5943	9.9
	(c) TRYCOL 6720	22.1
	(d) water	<u>60.0</u>
10		100.0

EXAMPLE 8

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
15	(a) GLUCOPON 600 UP	0.0
	(b) TRYCOL 5943	0.0
	(c) TRYCOL 6720	40.0
	(d) water	<u>60.0</u>
		100.0

EXAMPLE 9

A scouring and bleaching surfactant composition was prepared having the following components:

	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	0.0
25	(b) TRYCOL 5943	40.0
	(c) TRYCOL 6720	0.0
	(d) water	<u>60.0</u>
		100.0

EXAMPLE 10

A scouring and bleaching surfactant composition was prepared having the following components:

5	<u>COMPONENTS</u>	<u>%/wt.</u>
	(a) GLUCOPON 600 UP	40.0
	(b) TRYCOL 5943	0.0
	(c) TRYCOL 6720	0.0
	(d) water	<u>60.0</u>
10		100.0

EXAMPLES 1-10

Examples 1-10 were used to scour and bleach cotton substrates by impregnation with an aqueous peroxide bleaching bath having a pH of about 11.0, followed by steaming at a temperature of about 98°C for a period of about 60 minutes.

Each sample was evaluated per the above stated testing methods for whiteness, foam formation, alkali stability, wetting properties and cleanliness, i.e., % Extractibles, the results being set forth in Table I.

TABLE I

EXAMPLE	FOAM PRODUCED	WHITENESS RATING	DRAVES TEST (SEC.)	(%) EXTRAC- TIBLES	CAUSTIC STABILITY (% OF 50% NaOH)
1	high	76.12	6.4	1.01	60
2	very high	75.73	14.3	0.35	80
3	high	76.2	4.9	0.57	2.5
4	meduim	77.15	9.8	0.91	70
5	low	74.6	5.7	0.43	50
6	very high	75.1	11.3	0.41	70
7	high	75	11.3	0.68	50
8	very low	72.73	6.5	0.59	1
9	high	75.1	8.5	0.47	40
10	very high	71.82	14.5	0.54	99

From the foregoing evaluation results, it can be seen that the bleaching and scouring process of this invention synergistically provides excellent whiteness to fabric materials, low residual impurities, is stable in high concentrations of strongly alkaline materials at high temperatures, and is low-foaming under high agitation conditions.

What is claimed is:

1. A process for bleaching and scouring textile materials comprising adding to an aqueous alkaline peroxide bath an effective amount of a surfactant composition  
5 comprising:

(a) from about 94.0 to about 6.0% by weight of an alkyl polyglycoside having the general formula I



wherein R is a monovalent organic radical having from about  
10 8 to about 16 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6,

(b) from about 6 to about 94% by weight of a blend of alkoxyated branched C<sub>8</sub>-C<sub>15</sub> alcohols, and

15 (c) the remainder water, all weights being based on the weight of the composition;

contacting said textile materials with said bath and then steaming said textile materials.

2. The process of claim 1 wherein said alkyl  
20 polyglycoside has from about 12 to about 16 carbon atoms.

3. The process of claim 1 wherein said alkyl polyglycoside has a percent actives of about 50%.

4. The process of claim 1 wherein said alkoxyated alcohols contain from about 6 to about 12 moles of ethylene  
25 oxide and from about 4 to about 8 moles of propylene oxide per mole of said alcohol.

5. The process of claim 4 wherein said alkoxyated

alcohols have an HLB value of from about 6 to about 15.

6. The process of claim 1 wherein said blend of alkoxyated alcohols comprises a mixture of a tridecyl alcohol ethoxylated with about 12 moles of ethylene oxide per mole of alcohol and an isodecyl alcohol containing about 6 moles of ethylene oxide and about 6 moles of propylene oxide.

7. The process of claim 1 wherein said textile materials comprise cotton or a blend of polyester and cotton.

8. The process of claim 1 wherein from about 0.1 to about 1.0% by weight of said surfactant composition is added to said aqueous bath, based on the weight of said bath.

9. The process of claim 1 wherein from about 0.1 to about 0.2% by weight of said surfactant composition is added to said aqueous bath, based on the weight of said bath.

10. The process of claim 1 wherein said surfactant composition contains from about 20 to about 60% by weight of said component (a) and from about 80 to about 40% by weight of said component (b).

11. The process of claim 1 wherein said bath further contains from about 5.0 to about 20.0% by weight of additional finishing components, based on the weight of said bath.

12. The process of claim 11 wherein said finishing components are selected from sodium hydroxide, sodium silicate, and mixtures thereof.

13. The process of claim 1 wherein said bath is a bleaching exhaust bath.

14. The process of claim 1 wherein said bath is a scouring exhaust bath.

15. The process of claim 1 wherein said bath is both a scouring and bleaching exhaust bath.

5 16. The process of claim 1 wherein said bath is a bleaching padding bath.

17. The process of claim 1 wherein said bath is a scouring padding bath.

10 18. The process of claim 1 wherein said bath is both a scouring and bleaching padding bath.

19. The process of claim 15 wherein said scouring and bleaching exhaust bath is at a temperature in the range of about 180°F. to about 200°F.

15 20. The process of claim 1 wherein said bath has a pH of about 10.8 to about 11.2.

21. A surfactant composition for use in bleaching and scouring textile materials while controlling foam generation, said composition comprising:

20 (a) from about 94.0 to about 6.0% by weight of an alkyl polyglycoside having the general formula I



wherein R is a monovalent organic radical having from about 8 to about 16 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a  
25 value from about 1 to about 6,

(b) from about 6 to about 94% by weight of a blend of alkoxyated branched C<sub>8</sub>-C<sub>15</sub> alcohols, and

(c) the remainder water, all weights being based on the weight of the composition.

22. A composition as in claim 21 wherein said alkyl polyglycoside has from about 12 to about 16 carbon atoms.

5 23. A composition as in claim 21 wherein said alkyl polyglycoside has a percent actives of about 50%.

24. A composition as in claim 21 wherein said alkoxyated alcohols contain from about 6 to about 12 moles of ethylene oxide and from about 4 to about 8 moles of propylene oxide  
10 per mole of said alcohol.

25. A composition as in claim 21 wherein said alkoxyated alcohols have an HLB value of from about 6 to about 15.

26. A composition as in claim 21 containing from about 20% to about 60% by weight of said component (a) and from about  
15 80% to about 40% by weight of said component (b).

27. A composition as in claim 21 wherein said blend of alkoxyated alcohols comprises a mixture of a tridecyl alcohol ethoxylated with about 12 moles of ethylene oxide per mole of alcohol and an isodecyl alcohol containing  
20 about 6 moles of ethylene oxide and about 6 moles of propylene oxide.

28. A composition as in claim 21 further containing from about 5.0% to about 20.0% by weight of additional finishing components, based on the weight of said composition.

25 29. A composition as in claim 28 wherein said finishing components are selected from sodium hydroxide, sodium silicate, and mixtures thereof.



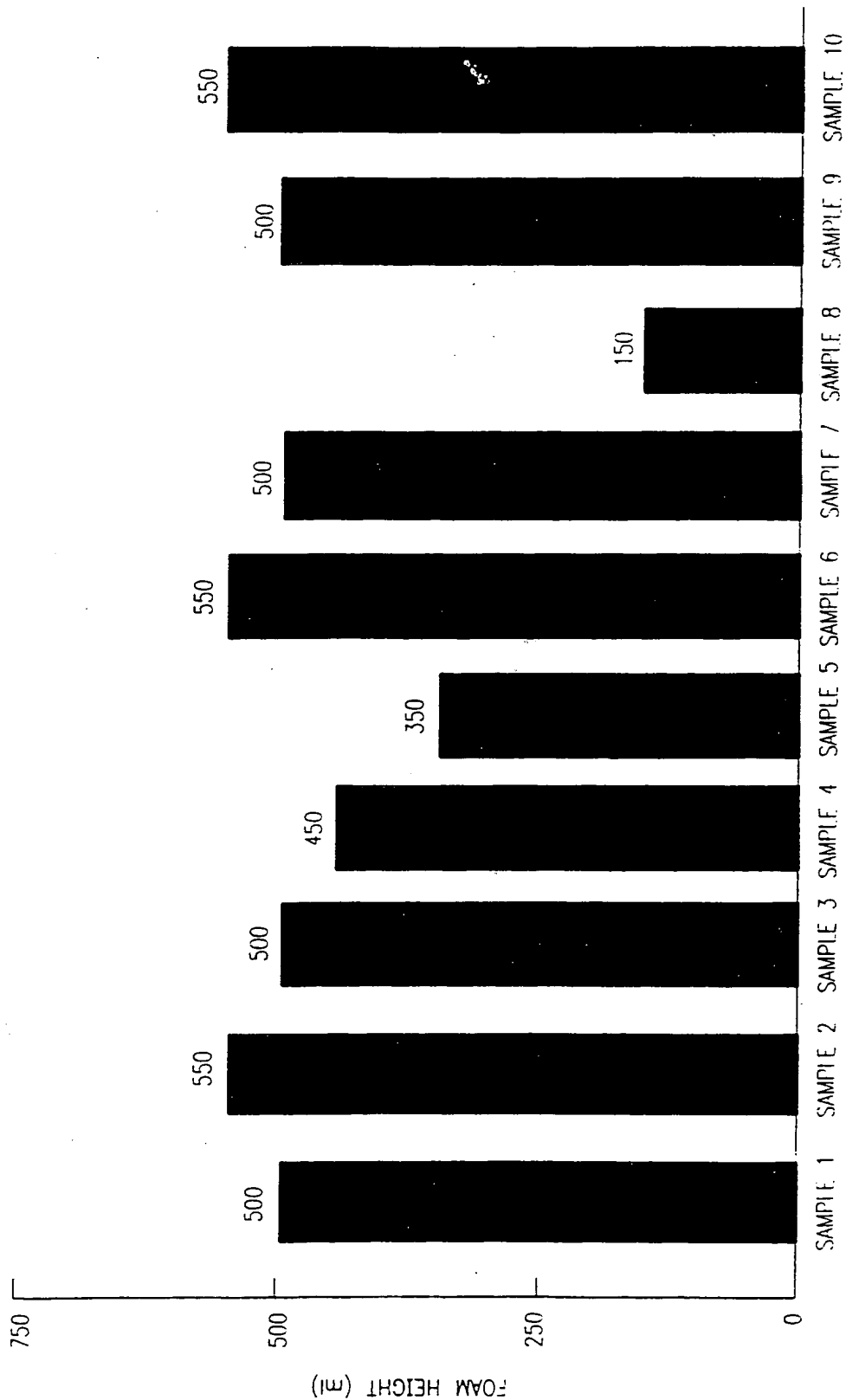


FIG. 1

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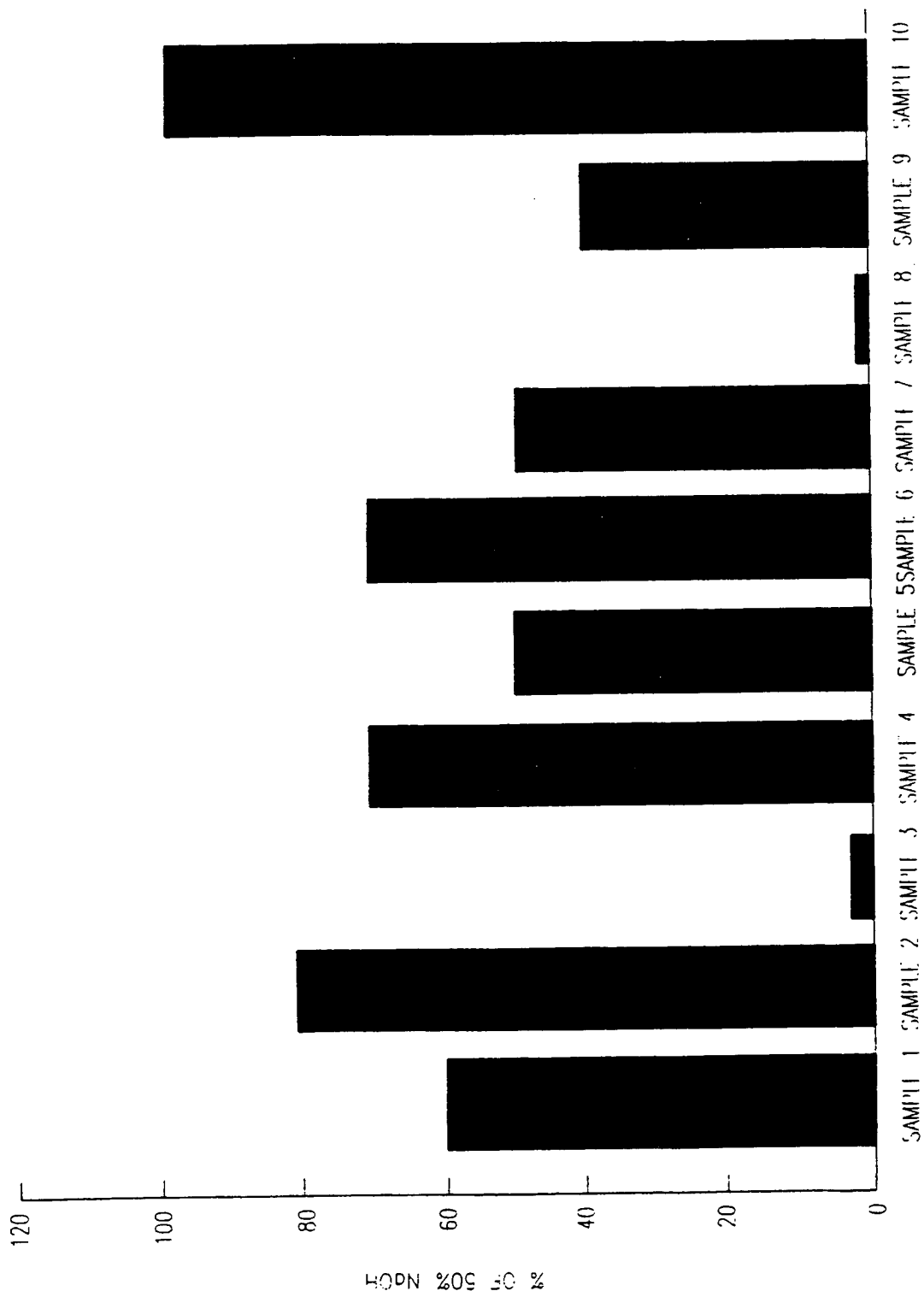


FIG. 2

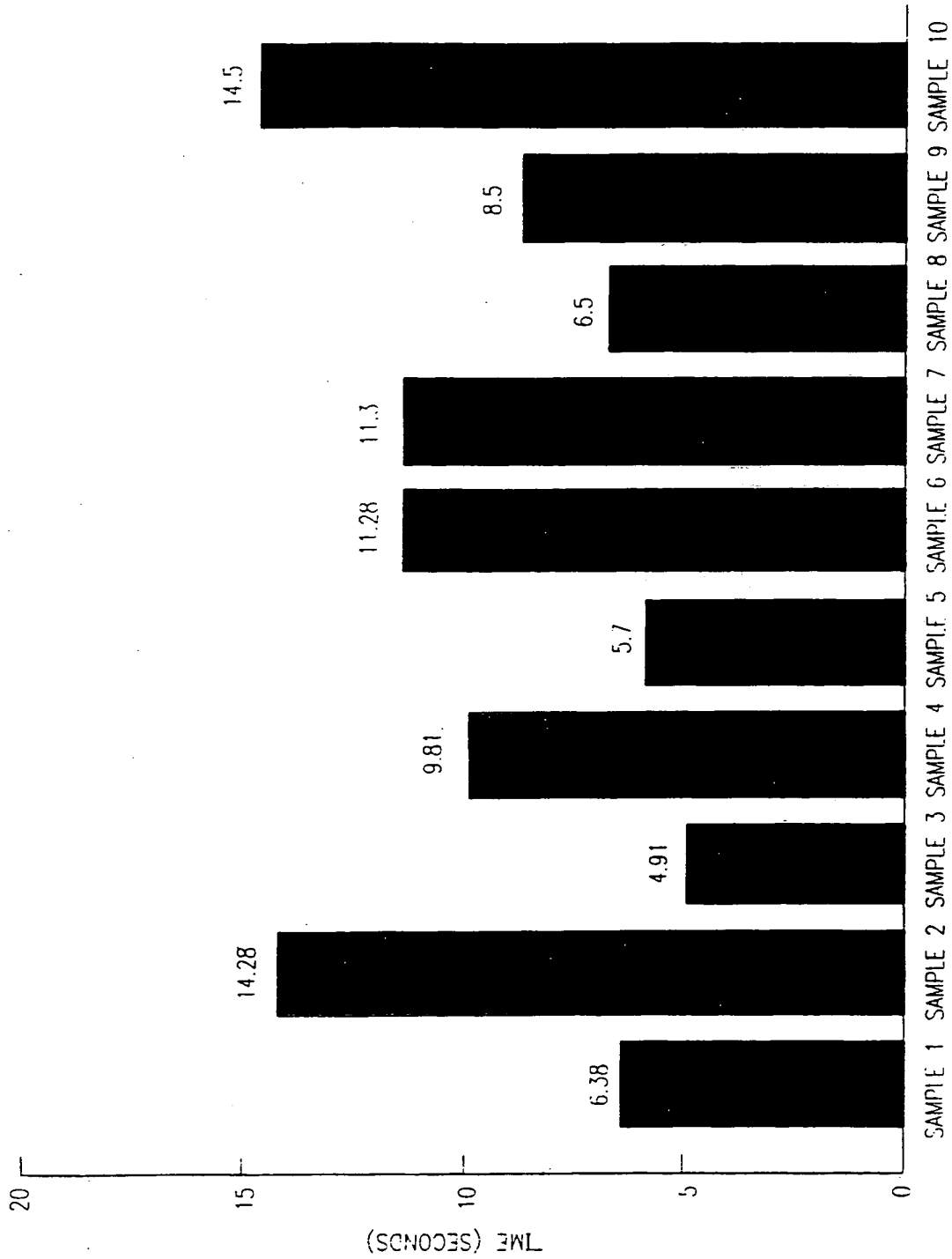


FIG. 3

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## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US98/08585

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :D06L 3/02; C11D 1/66, 1/72, 1/72

US CL :8/111, 139; 510/535, 303, 470, 506, 340; 252/351

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 8/111, 139; 510/535, 303, 470, 506, 340; 252/351

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4,483,780 A (LLENADO) 20 November 1984 (20-11-84), column 1, lines 37-57; column 2, lines 50-68; column 5, lines 5-35; and column 6, lines 1-38.	21-23, 25, 26, 28, 29
X	US 5,047,168 A (BROZE et al) 10 September 1991 (10-09-91), column 5, line 61 through column 6, line 65; column 8, lines 10-24; column 9, lines 49-68; and column 12, lines 1-41.	21-26, 28, 29
A	US 5,527,362 A (COLE et al) 18 June 1996 (18-06-96).	1-29
A	US 5,542,950 A (COLE et al) 06 August 1996 (06-08-96).	1-29



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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*L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*A* document member of the same patent family
*O* document referring to an oral disclosure, use, exhibition or other means	
*P* document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

29 MAY 1998

Date of mailing of the international search report

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